EXHIBIT A

PATENT
Application No. 09/868,379
Filing Date: 08/15/2001
Examiner: Michelle Graffeo
Art Unit: 1614
Attorney Docket No. H03763 PCT/US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the United States Patent Application of
Applicants: Christian Kropf,
Ulrike Brüninghaus,
Amerigo Pastura,
Michael Meinders,
Peter Wölknitz,
Rolf Hempelmann and
Marcel Roth

Application Serial No. 09/868,379
Filing Date: 08/15/2001
Claiming priority of International Application
PCT/EP99/09683, filed 12/09/1999
and German Application
DE 198 53 662.0, filed 12/18/1998

Examiner: Michelle Graffeo
Art Unit: 1614

Assignee: Henkel KGaA

Title: **FINE SUSPENSIONS OF POORLY SOLUBLE CALCIUM SALTS AND THEIR USE IN DENTAL CARE PRODUCTS**

DECLARATION OF CHRISTIAN KROPF

I, Christian Kropf declare as follows:

1. I am an inventor of United States Patent Application No. 09/868,379.

2. I am head of a department in Corporate Research Chemistry at Henkel KGaA, Henkelstraße 67, 40589 Düsseldorf, Germany. I obtained both a diploma degree in Chemistry in 1992 and a Ph.D. degree in Engineering Sciences (new materials) in 1998 from Saarland University in Saarbruecken, Germany.

3. I am familiar with United States Patent Application No. 09/868,379 of Christian Kropf et al. (hereinafter the "Kropf application"), the S. Zhang et al. publication, J. Mater. Sci. Mater., Med. 8 (1997), 25–28 (hereinafter the "S. Zhang publication") and United States Patent No. 5,741,773 to Y.P. Zhang et al. (hereinafter the "Y.P. Zhang patent").

4. Claim 8 of the Kropf application is directed to a suspension. The remaining claims 9–21 are directed to a process of preparing the suspension, a method of remineralizing teeth, toothpaste comprising the suspension, or other suspensions, all of which comprise the suspension of claim 8 or a more narrowly defined suspension. Claim 8 reads as follows:

> A suspension of one or more phosphate calcium salts, fluoride calcium salts or fluorophosphate calcium salts in a liquid medium in which the salts are less than 1 g/l soluble, wherein the calcium salts comprise primary particles having diameters of from 5 to 50 nanometers and lengths of from 10 to 150 nanometers, stabilized against agglomeration by a content of at least 0.01% by weight, based on the weight of the suspension, of a water-soluble surfactant or of a natural water soluble polymeric protective colloid selected from the group consisting of gelatin, casein, albumin, starch, plant gums, water-soluble derivatives of water-insoluble natural polymeric substances, cellulose esters, methylcellulose, hydroxyethylcellulose, carboxymethylcellulose, hydroxyethylstarch and hydroxypropyl guar, adsorbed onto said particles.

5. The S. Zhang publication discloses a new method of preparing thermally stable, pure, rod-like HAp (hydroxyapatite) particles. (Page 25, first column, lines 25–28). According to the method disclosed in the S. Zhang publication, polyacrylic acid is added to a solution to obtain a precipitation of particles of pure hydroxyapatite. Instead of particles comprising both hydroxyapatite and tricalcium phosphate. This reason for using a solution comprising polyacrylic acid in Zhang's method of preparing pure hydroxyapatite particles is disclosed, for example, in the S. Zhang publication in the following passage bridging pages 26 and 27:

> A pure HAp well-crystallized structure (Fig. 2e, SH-2) is obtained from the system containing polyacrylic acid and treated hydrothermally. A well-crystallized structure is also observed in the sample S-2, however, it is a mixture of TCP (tricalcium phosphate) and HAp (Fig. 2d, S-2). ***Therefore, hydrothermal treatment and polyacrylic acid addition stabilized the synthetic hydroxyapatite.*** (*emphasis added*).

This purpose for using polyacrylic acid is also illustrated by the following statement in the S. Zhang publication bridging pages 27–28:

> Fig. 4a is a typical XPS core spectrum for Ca2p which has 2 peaks separated by 3.5 eV in bonding energy. Another calcium peak appeared at low binding energy, with area approximately 20% of the total calcium element, which matches the amount of PAA added. This calcium is bonded to polyacrylic acid [13, 14]. A weight loss of 20% also appeared in the TGA curve in the temperature range 300–400°C. ***The detailed mechanism of how the polyacrylic acid stabilizes the hydroxyapatite during calcining up to 1100°C is under further investigation.*** (*emphasis added*).

6. The S. Zhang publication also discloses a process of obtaining precipitated HAp, not a suspension of HAp in a liquid. The precipitated HAp produced according to the process disclosed in the S. Zhang publication is in the form of particles consisting of rod-like crystals. (Fig. 1 and page 26, second column, lines 1–8).

7. In the S. Zhang publication, the statement in the caption of Fig. 1 and in the last sentence of column 1, page 26: "Here 1 represents the system without polyacrylic acid and 2 the system with polyacrylic acid in the starting precipitates" refers to hydrothermally treated precipitates which were made with solutions containing the starting materials with or without polyacrylic acid. Figs. 1(a) and (b) are identified as showing "TEM photographs of rod-like crystals of HAp after hydrothermal treatment: (a) without polyacrylic acid (H-1); and (b) with polyacrylic acid in the system (H-2)." The "TEM" (transmission electron microscope, operated under high vacuum conditions) photographs show precipitated and hydrothermally treated crystalline particles of HAp arranged on a metal grid (typically copper or nickel) with an amorphous carbon or formvar® layer as a specimen carrier, and do not show Applicants' claimed suspension comprising calcium salt nanoparticles in a liquid.

8. The S. Zhang publication also discloses that the particles precipitated in the solution comprising polyacrylic acid, which is a synthetic colloid, produce poorly crystallized apatite structures. Such poorly crystallized apatite structures must be further treated hydrothermally and then sintered or calcined at 1,100°C for two hours to make pure HAp well-crystallized particles. (Page 26, second column, line 19, to page 27, column 1, line 1). These HAp particles contain no polyacrylic acid because all polyacrylic acid that was present in the system used to prepare the precipitated HAp particles is decomposed and evaporated during the calcining step. The pure HAp is shown by SH-2 in Figs. 2(f) and 3.

9. The calcium particles claimed in claim 8 of the Kropf application are not pure HAp. They are particles of calcium salt with a colloid selected from a group of natural colloids adsorbed onto said particles. Accordingly, the particles claimed in the Kropf application are different in composition than the pure HAp particles produced after the sintering process step disclosed by the S. Zhang publication.

10. A comparison of the limitations set forth in claim 8 with the subject matter disclosed in the S. Zhang publication reveals that the claimed suspension is not taught or suggested by the S. Zhang publication. The S. Zhang publication discloses the production of particles of pure HAp, or a mixture of HAp and TCP (tricalcium phosphate) (as shown in Fig. 3). Accordingly, the S. Zhang particles are therefore distinct from Applicants' claimed particles of calcium salt, wherein a natural water-soluble polymeric protective colloid is adsorbed onto said particles. The Y.P. Zhang patent does not disclose calcium salt particles in the form of nanoparticles. Accordingly, the S. Zhang publication and the Y.P. Zhang patent do not disclose or suggest Applicants' claimed suspension comprising particles of calcium salt with a colloid adsorbed onto said particles, which is set forth in all of Applicants' pending claims 8–21.

11. If the Y.P. Zhang patent and the S. Zhang publication are considered in their entireties, including disclosures that teach away from the claims, the Y.P. Zhang patent teaches away from the claimed suspension of calcium particles in the form of nanoparticles in a liquid. The Y.P. Zhang patent does not even disclose nanoparticles. The S. Zhang publication discloses the production of solids of pure rod-like HAp particles, or a mixture of HAp and TCP particles (as shown in Fig. 3), which is distinct from Applicants' claimed liquid suspension of particles of calcium salt, wherein a surfactant or natural water-soluble polymeric protective colloid is adsorbed onto said particles. The Y.P. Zhang patent and the S. Zhang publication, therefore, fail to disclose Applicants' claimed suspension.

12. An effort to modify the process disclosed in the S. Zhang publication to try to obtain Applicants' claimed particles renders the S. Zhang process unsatisfactory for its intended purpose. More specifically, eliminating the sintering step from the process disclosed in the S. Zhang publication to try to incorporate polyacrylic acid onto the HAp particles renders the process unsatisfactory for its purpose of creating pure crystalline hydroxyapatite particles.

13. The principle of operation of the S. Zhang publication is the production of precipitated solids of pure HAp particles (as shown in Fig. 3). Accordingly, the particles are distinct from Applicants' claimed suspension comprising particles of calcium salt, wherein a natural water-soluble polymeric protective colloid is adsorbed onto said particles. A modification of the process disclosed in the S. Zhang publication, eliminating the hydrothermal and sintering treatment steps in an effort to try to obtain Applicants' claimed suspension, would change the principle of operation of the reference, as those steps are required by S. Zhang to obtain pure HAp well-crystallized particles.

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 19 of the United States Code, and that such willful false statements may jeopardize the validity of the Kropf application or any patent issued thereon.

Dated: September 1, 2006

CHRISTIAN KROPF